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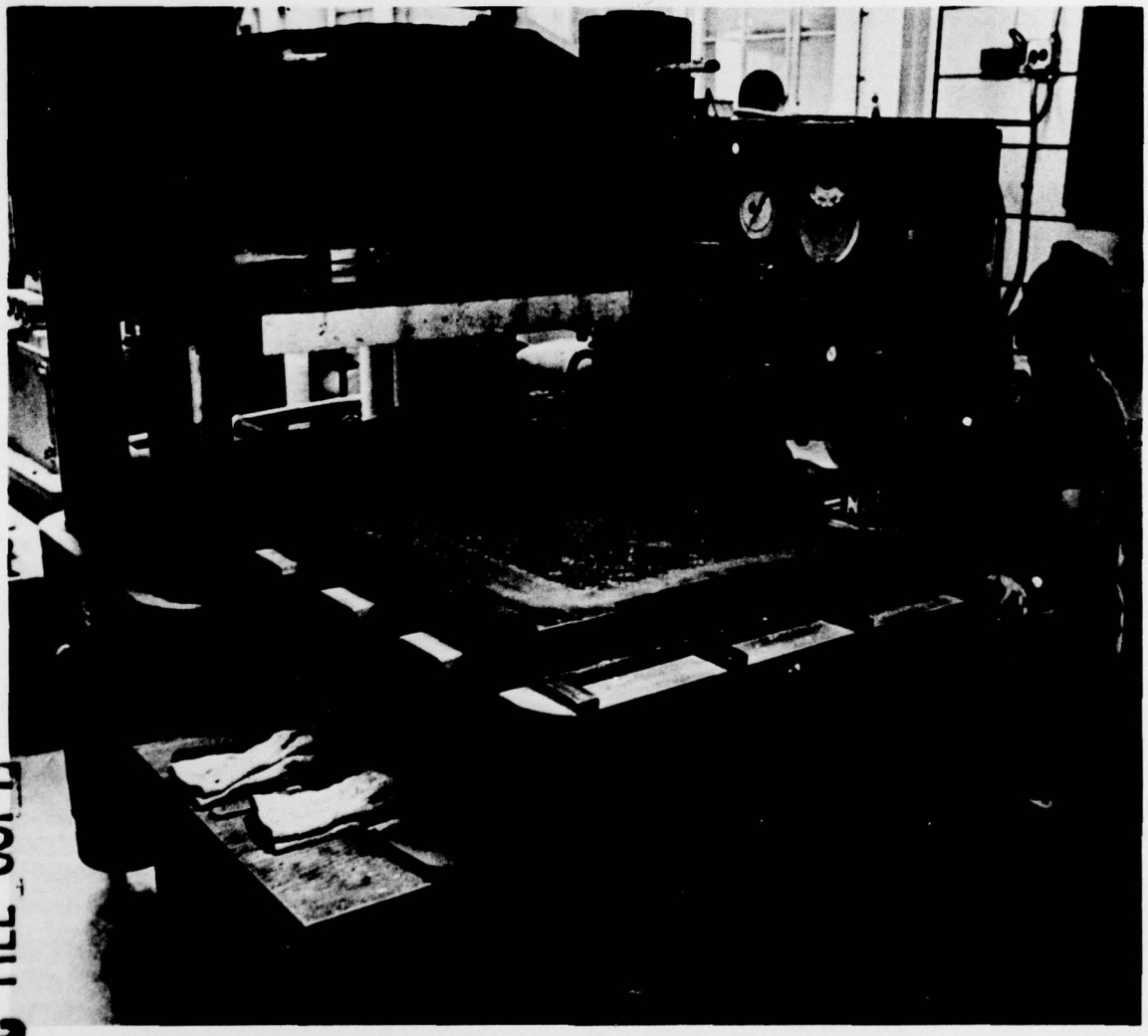

Abstract

Wood fiber for dry-formed hardboard was refined under pressure from red oak and balsam fir. The fiber of each species was adjusted to four acidity levels before treating with two phenolic resins, an alkaline curing and an acid curing, that differed in degree of polymerization. An optimum fiber acidity at which most strength properties reached their maximum was found for each of the two resins. By adjusting fiber acidity, red oak boards had strength properties comparable to balsam fir boards. Boards from various blends of the red oak and the balsam fir fiber adjusted to a common pH value possessed strengths of comparable values. Linear and thickness stability were more dependent on species than were other properties. Acidity control was found beneficial in maintaining board quality.

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HOW FIBER ACIDITY AFFECTED FUNCTIONAL PROPERTIES OF DRY-FORMED HARDBOARDS.

By

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Introduction

Dry-formed hardboards are generally produced from a single species of wood or a controlled mixture of a limited number of species. Phenolic resin binder systems are selected for their suitability for the wood supply and processing conditions. Board quality has been more difficult to maintain with a heterogeneous wood supply. With an increasing cost of wood and a decreasing supply of good quality raw material, the hardboard industry will have to rely increasingly on residues and wood mixes less homogeneous in nature. The mixes will consist of whole tree chips, wood residues from logging, and wood wastes of urban areas. If we are to meet future demands for wood, these types of materials must be utilized more fully.

Fiber acidity varies between species, within species, between heartwood and sapwood, and between young trees and old trees (*4*).[2] Acidity increases if wood is stored under damp conditions and is accelerated if wood is heated (*3, 7*). The extractives in many woods, especially heartwoods, are highly acidic and have a high buffering capacity (*8*). Species and acidity differences are minimized in manufacturing wet-formed hardboards by adjusting pH to a specific level to retain resin and fix size on fiber. Recent studies indicate a possibility to improve dry-formed hardboard properties by controlling pH (*6, 9*). Nelson (*6*) found, with a limited number of species, that strength properties correlated with pulp acidity. Adjusting pH could be a practical approach to increase the utilization of forest residues in reconstituted wood fiber products.

The objectives of this investigation were to determine: (1) If there is an optimum pulp acidity for wood-resin interaction; (2) how acidity is affected by a particular resin system; and (3) if control of pulp acidity offsets species differences.

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[1] Maintained at Madison, Wisconsin, in cooperation with the University of Wisconsin.

[2] Italicized numbers in parentheses refer to literature cited at end of report.

Materials and Methods

Wood

Two species of wood were used in this study, northern red oak (*Quercus rubra L.*) and balsam fir (*Abies balsamea* (*L.*) *Mill.*). Red oak is a short-fibered, high-density hardwood; balsam fir is longer fibered, and is a low-density softwood. The acetyl contents of these woods were 4.1 percent and 1.6 percent, respectively.

Pulping

Pulpwood logs were peeled and converted into nominal 1/2-inch chips by a commercial-size four-knife chipper.

Pulping was in an Asplund defibrator. The schedule used included presteaming under full pressure (175 lb/in.^{2}) for 1 minute, primary refining under full pressure (175 lb/in.^{2}) for 3 minutes, then gradually dropping the pressure to 100 pounds per square inch and holding for 5 minutes during secondary refining.

To retain all of the water-soluble extractives, water was not used to wash the pulp from the mill. The pulp remaining in the mill after blowdown was added to the fibers collected in the cyclone. The wet pulp was air dried and lightly fluffed in an 8-inch disk refiner equipped with knobby plates to break down large clusters of fibers.

Adjusting Fiber Acidity and Treating with Resin

The air-dried fibers were adjusted to one of four acidity levels (3.5, 4.5, 5.5, or 6.5) by spraying them with a buffering solution while they were tumbled in a revolving drum. The buffering solution was either a 1- or 2-percent solution of sulfuric acid or a 5- or 10-percent solution of sodium bicarbonate depending on the direction, up or down, the adjustment was to be made.

Two phenolic resin types commonly used in manufacturing hardboards were used. A neutral pH, acid-curing low-advanced (polymerization) phenolic resin (A) was diluted to 32.5 percent concentration and the pH lowered to 4 with acetic acid. An alkaline-curing, 9.7 pH, medium-advanced (polymerization) phenolic resin (B) was diluted to 25 percent concentration. The fiber was treated with 8 percent resin solids, based on ovendried weight of fiber and chemical, by spraying the dilute resin onto the fiber as it was tumbled in a revolving drum. After resin application, the fiber was tumble dried to 8 to 10 percent moisture content in a modified clothes dryer.

Boardmaking

Three medium-density (nominal 45 lb/ft^3) hardboards of 3/8-inch thickness and three high-density (nominal 60 lb/ft^3) hardboards of 1/8-inch thickness were made from the individual species, the red oak and from the balsam fir, and from fiber blends of the two species. Mats were formed on a 14- by 14-inch banjo former. The forming box had filaments strung at 1/4-inch intervals in both directions. The fiber was placed on this network, and the filaments vibrated; this caused the fibers to pass through the network onto a caul. The mats were cold-pressed, then hot-pressed for 10 minutes at 374° F for the medium-density boards and for 6 minutes at 374° F for the high-density boards. Stops placed between the cauls controlled thickness. All boards were heat treated for 1 hour at 325° F after hot pressing.

Evaluations

Fiber acidity was determined by placing 1 gram of dry fiber in 5 grams of boiled distilled water and measuring pH with an electronic meter (*5*). Fiber acidity was determined after (a) air drying, (b) adjusting acidity, (c) applying resin, (d) drying the fiber before boardmaking, and (e) curing and heat treating the boards. Before determining acidity of the boards they were milled to a flour.

All boards were preconditioned for at least 30 days at 50 percent relative humidity and 73° F. Flexural strength, tension parallel to surface (tensile strength), and tension perpendicular to the surface (internal bond) were determined according to ASTM method D 1037-72a (*1*) on two specimens per panel. Modulus of rupture and modulus of elasticity were calculated from the flexural test data.

To determine dimensional stability, 1/2- by 6-inch specimens were conditioned at 50 percent relative humidity (RH) for 30 days, then exposed to several conditions. For the dimensional stability, length, thickness, and weight changes were determined after exposure to 90 percent RH and 80° F for 30 days, after immersion in water for 30 days, and ovendrying at 220° F for 72 hours.

Discussion of Results

Strength properties and dimensional movement of the medium-density hardboard made from balsam fir and from red oak, of the high-density hardboards from red oak, and of the medium-density hardboards from blends of the balsam fir and red oak are presented in tables 1 through 3. Included in these tables are coefficients of variation, some of which show considerable variation. This variability is probably caused by board formation, resin, and wood species, either individually or in combinations with each other.

The variations were greater with resin A than with resin B. Apparently resin penetration into the fiber was less uniform with resin A than B because of their differences in degree of polymerization; this caused differences in bonding; subsequently board results varied.

More variation was also noted in hardboard from red oak than in that from balsam fir, and was attributed to the more complex anatomical nature of the red oak.

Properties of Hardboards from Single Species

The properties of the medium-density hardboards from red oak and those from balsam fir are presented in table 1. The strength values have been adjusted to a common density of 42 pounds per cubic foot by dividing actual strength by actual density and multiplying the product by 42 pounds per cubic foot.

The acidity of the pulp fiber when the resin was applied influenced hardboard strength; however, the optimum acidity level was dependent on the type of phenolic resin used as the binder. For acid-curing resin A, the most satisfactory overall properties were usually achieved at a fiber pH of 3.5; for alkaline-curing resin B, however, a fiber pH of 5.5 was most effective. Bending strength and stiffness were affected more by changes in fiber acidity than was tensile strength, and the changes in tensile strength were more noticeable with the oak boards than with the balsam fir boards. Internal bond strength did not always follow the same pattern as the other strength properties.

Before any acidity adjustment, the oak and balsam fir pulps had pH values of 3.5 and 4.7, respectively. The oak pulp was already at the desirable acidity level for the acid-curing phenolic, but for high-quality boards with the alkaline-curing resin the pulp pH had to be adjusted to about 5.5. To achieve the highest strength for the balsam fir boards, the alkalinity of the pulp with the alkaline-curing resin had to be slightly increased as did the acidity for the acid-curing resin. By adjusting fiber acidity it was possible to make oak boards comparable in strength to balsam fir boards.

Dimensional movement and water absorption of the medium-density boards are presented in table 1. Linear and thickness stability (50 to 90 pct RH) were not affected as much by changes in fiber acidity as were strength properties. Regardless of the resin used, the boards made with fiber at 3.5 pH were more stable than those made with fiber at 6.5 pH. This was contrary to Nelson's finding that linear stability was related to fiber acidity.(*6*).

The balsam fir board surpassed the oak in linear stability but had greater thickness expansion. In scanning electron micrographs of the boards at our Laboratory, the balsam fir fibers appeared flatter than did those of the oak. This would be expected since balsam fir is less dense than oak and requires more compression for a given density board.

Boards made with acid-curing resin A were more dimensionally stable than boards with alkaline-curing resin B. Resin A is a lower molecular weight resin with greater penetrating characteristics than B, whereas B is more of a bonding-type resin and provides less resistance to moisture entering the fiber. These findings confirm those reported by Fahey and Pierce for a two-step resin treatment (*2*).

High-density hardboards were made with only bonding resin B. The property values (table 2) were somewhat similar to those of the medium-density hardboards. However, maximum bending and tensile properties were attained at a fiber pH of 4.7 instead of 5.5 as noted with the medium-density hardboard. This shift was apparently due to increased hydrolysis caused by higher pressures and temperatures within the board in making the high-density board.

Properties of Hardboards from Wood Blends

The strength properties and dimensional movement determined for medium-density hardboards made from blends of oak and balsam fir are given in table 3. Included also are the 100-percent balsam fir and the 100-percent oak boards made from fibers that had been

Table 1. -- Strength properties and dimensional movement of medium-density hardboard from balsam fir and red oak[1]

Fiber type	Fiber pH before resin	Bending[2]		Internal bond maximum stress[2]	Tensile strength maximum stress[2]	Length change[3] from 50 percent relative humidity to --		Thickness change[3] from 50 percent relative humidity to --	
		Modulus of rupture	Modulus of elasticity			90 per cent relative humidity	Water soaked	90 per-cent relative humidity	Water soaked
		Lb/in.²	1,000 lb/in.²	Lb/in.²	Lb/in.²	Pct	Pct	Pct	Pct
				ACID-CURING PHENOLIC RESIN A					
Balsam fir	3.5	2,800 (9.04)	384 (6.77)	31 (32.26)	1,320 (8.03)	0.22 (4.55)	0.33 (6.06)	4.97 (5.43)	8.63 (1.39)
	4.6	2,490 (18.88)	340 (14.41)	34 (8.82)	1,330 (7.07)	.23 (4.35)	.30 (10.00)	5.86 (5.46)	9.88 (5.67)
	5.5	2,520 (12.22)	322 (8.70)	40 (25.00)	1,370 (10.80)	.25 (8.00)	.32 (6.25)	5.16 (6.01)	9.22 (4.34)
	6.6	2,310 (14.29)	319 (10.03)	27 (25.93)	1,300 (3.46)	.24 (4.17)	.30 (3.33)	6.46 (5.11)	10.04 (1.59)
Red oak	3.5	2,230 (34.39)	327 (32.72)	58 (17.24)	1,260 (5.40)	.51 (0)	.72 (1.39)	3.89 (.51)	6.67 (.45)
	4.5	2,190 (18.40)	326 (13.80)	59 (38.98)	960 (6.77)	.58 (1.72)	.76 (2.63)	3.93 (.51)	6.37 (1.88)
	5.4	1,940 (7.99)	331 (5.74)	17 (52.94)	920 (11.30)	.59 (0)	.69 (1.45)	4.10 (4.15)	6.43 (4.51)
	6.5	1,810 (22.93)	284 (23.59)	35 (31.43)	900 (17.33)	.62 (0)	.79 (2.53)	4.29 (3.73)	6.79 (4.71)
				ALKALINE-CURING PHENOLIC RESIN B					
Balsam fir	3.6	2,670 (10.52)	338 (6.51)	28 (10.71)	1,390 (2.59)	.32 (6.25)	.36 (5.56)	6.32 (4.59)	10.60 (4.15)
	4.4	2,880 (10.49)	359 (7.24)	43 (6.98)	1,180 (11.10)	.35 (2.86)	.43 (2.33)	4.76 (4.20)	8.33 (8.40)
	5.5	2,950 (6.31)	364 (5.77)	32 (12.50)	1,390 (7.55)	.33 (6.06)	.40 (7.50)	5.45 (8.26)	9.35 (7.91)
	6.7	2,400 (14.29)	303 (10.23)	38 (26.32)	1,340 (5.07)	.36 (2.78)	.40 (2.50)	6.69 (2.54)	10.87 (5.24)
Red oak	3.2	2,180 (13.94)	356 (7.58)	24 (29.17)	860 (6.51)	.67 (1.49)	.89 (1.12)	3.50 (4.86)	5.05 (16.44)
	4.6	2,130 (7.93)	289 (4.50)	44 (9.09)	730 (24.11)	.77 (1.30)	.97 (2.06)	4.11 (3.65)	6.75 (4.00)
	5.6	2,920 (10.89)	392 (10.46)	48 (20.83)	1,040 (16.54)	.77 (1.30)	.95 (2.11)	3.71 (4.31)	5.78 (3.29)
	6.7	2,510 (7.81)	347 (8.65)	34 (14.71)	870 (12.87)	.79 (2.53)	.98 (2.04)	4.10 (3.90)	6.06 (12.05)

[1]Values in parentheses are coefficients of variations and represent single specimens.

[2]Values are average of 6 specimens.

[3]Values are average of 3 specimens.

adjusted to the same acidity level. Although some variations in strength properties between the different blends were noted, the bending and the tensile strength values generally fell within the 95-percent confidence limits. The initial acidity levels for these species blends varied from 4.4 pH for the mixture with 75 percent balsam fir to 3.6 pH for the mixture with 75 percent oak. With acidity adjusted to the optimum for a particular resin, some variation in wood composition can be accommodated.

Strength was easier to maintain than was linear stability. Addition of 25 percent oak to the balsam fir board had little, if any, effect on linear change related to change in relative humidity from 50 to 90 percent. However, the boards with 75 percent oak and those with 100 percent oak exhibited considerably greater linear movement but less thickness expansion than did the boards with high balsam fir content. Only a slight reduction in dimensional movement occurred after three cycles between 50 and 90 percent relative humidity.

Adjusting Fiber Acidity Level

Obviously a number of times are available during pulp preparation to adjust acidity. In preliminary tests on the boards, the following three possible times when acid or alkali could be added were investigated: (1) When the chips entered the defibrator, (2) when the fiber was partially refined in the defibrator, and (3) when the air-dried fiber was ready for resin treatment.

The most successful results were obtained when the chemical was sprayed onto the air-dried fiber. At this time and by this method it was also easiest to attain the desired acidity level. The lowest strength boards were obtained when the acid or the alkali was metered into the defibrator after partial fiberization. Although the fibers were somewhat separated, they were saturated with water; thus, at a high-temperature level, they were more vulnerable to degradation. Adding the chemical when the chips entered the defibrator affected hydrolysis and pulp strength was reduced.

Table 2. -- Strength properties and dimensional movement of high-density hardboard from red oak[1]

Fiber pH before resin	Bending[2]		Internal bond maximum stress[2]	Tensile strength maximum stress[2]	Length change[3] from 50 percent relative humidity to --		Thickness change[3] from 50 percent relative humidity to --	
	Modulus of rupture	Modulus of elasticity			90 percent relative humidity	Water soaked	90 percent relative humidity	Water soaked
	Lb/in.^{2}	1,000 lb/in.^{2}	Lb/in.^{2}	Lb/in.^{2}	Pct	Pct	Pct	Pct
				ALKALINE-CURING PHENOLIC RESIN B				
3.5	4,340 (22.44)	530 (22.26)	259 (43.24)	1,930 (18.29)	0.65 (4.62)	0.87 (3.45)	3.31 (1.51)	6.36 (8.49)
4.7	5,060 (16.66)	658 (13.07)	206 (32.04)	2,310 (14.85)	.68 (4.41)	.90 (3.33)	3.68 (16.58)	6.75 (1.93)
5.4	4,290 (13.66)	540 (16.30)	279 (35.13)	1,910 (12.36)	.65 (3.08)	.83 (1.20)	4.54 (10.79)	6.94 (7.64)
6.4	4,290 (20.30)	514 (16.93)	238 (35.71)	1,930 (16.63)	.69 (4.35)	.88 (4.55)	4.23 (13.24)	7.14 (8.40)

[1]Values in parentheses are coefficients of variations for single specimens.
[2]Values are average of 6 specimens.
[3]Values are average of 3 specimens.

Acidity Changes During Boardmaking

Although differences were large in pH value for the fibers used in making the boards, they were not as noticeable in the final medium-density hardboards. Treating the oak and the balsam fir fibers in the 3.5 to 5.5 pH range with the alkaline resin raised pH to almost neutral or slightly alkaline. During hot pressing, however, pH dropped to an average 5.85 apparently due to development of acetyl groups. The acid-curing phenolic resin had little if any effect on the pH of the fiber, and the

Table 3. -- Strength properties and dimensional movement of medium-density hardboard from fiber blends[1]

Fiber composition		Fiber pH	Bending[2]		Internal bond maximum stress[2]	Tensile strength maximum stress[2]	Length change[3] from 50 percent relative humidity to --		Thickness change[3] from 50 percent relative humidity to --	
Balsam fir	Red oak	before resin	Modulus of rupture	Modulus of elasticity			90 percent relative humidity	Water soaked	90 percent relative humidity	Water soaked
Pct	Pct		Lb/in.²	1,000 lb/in.²	Lb/in.²	Lb/in.²	Pct	Pct	Pct	Pct
					ACID-CURING PHENOLIC RESIN A					
0	100	3.5	2,230 (34.39)	327 (32.72)	58 (17.24)	1,260 (5.40)	0.51 (0)	0.72 (1.39)	3.89 (0.51)	6.67 (0.45)
25	75	3.6	2,560 (15.23)	388 (14.95)	25 (44.00)	920 (15.98)	.40 (5.00)	.62 (6.45)	3.59 (8.08)	6.63 (4.22)
50	50	3.45	2,850 (15.02)	416 (10.10)	14 (50.00)	1,270 (15.12)	.28 (7.14)	.42 (7.14)	3.69 (8.67)	7.56 (8.07)
75	25	3.4	2,790 (8.49)	424 (4.25)	13 (46.15)	1,240 (17.42)	.22 (0)	.32 (3.13)	3.87 (.52)	7.51 (4.53)
100	0	3.5	2,800 (9.04)	384 (6.77)	31 (32.26)	1,320 (8.03)	.22 (4.55)	.33 (6.06)	4.97 (5.43)	8.63 (1.39)
					ALKALINE-CURING PHENOLIC RESIN B					
0	100	5.6	2,920 (10.89)	392 (10.46)	48 (20.83)	1,040 (16.54)	.77 (1.30)	.95 (2.11)	3.71 (4.31)	5.78 (3.29)
25	75	5.4	2,340 (16.11)	362 (14.64)	17 (17.65)	990 (14.44)	.57 (5.26)	.76 (1.32)	3.97 (7.81)	7.41 (2.56)
50	50	5.5	2,810 (10.96)	406 (7.39)	14 (14.29)	1,120 (9.38)	.47 (6.38)	.61 (8.20)	4.43 (.68)	7.01 (2.14)
75	25	5.6	3,240 (7.16)	432 (6.48)	17 (11.76)	1,360 (11.18)	.36 (2.78)	.44 (2.27)	4.76 (3.57)	8.98 (1.56)
100	0	5.5	2,950 (6.31)	364 (5.77)	32 (12.50)	1,390 (7.55)	.33 (6.06)	.40 (7.50)	5.45 (8.26)	9.35 (7.91)

[1]Values in parentheses are coefficients of variations for single specimens.
[2]Values are average of 6 specimens.
[3]Values are average of 3 specimens.

pH did not change appreciably during pressing of the mats with 5.5 pH. However, during pressing of the 3.5 pH fiber mats, board pH increased to an average of 4.3. Because of the small differences in board pH, these medium-density boards should have comparable aging characteristics.

Conclusions

Based on the findings of this investigation, variations in hardboard strength caused by difference in mix of species can be minimized by controlling fiber acidity. Optimum acidity level is dependent on the phenolic resin binder. Linear stability and thickness stability, however, are more dependent on wood composition than on fiber acidity. These properties are improved by increasing acidity. For most satisfactory overall board properties, the acidity adjustment should be made on the air-dried fiber just before it is resin treated rather than during pulping and fiberizing.

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How fiber acidity affected functional properties of dry-formed hardboards, by Gary C. Myers. Madison, Wis. FPL, 1977.
9 p. (USDA For. Serv. Res. Pap. FPL 282).

Wood fiber of red oak and balsam fir for dry-formed hardboard is adjusted to four acidity levels before treating with an acid-curing and an alkaline-curing resin before testing board strength properties.

KEYWORDS: Red oak, balsam fir, wood fiber, fiber acidity, acid-curing phenolic, alkaline-curing phenolic, dry-formed hardboard, strength properties.

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